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(21) International Application Number: PCT/US97/15122 (22) International Filing Date: 28 August 1997 (28.08.97) (30) Priority Data: 60/024,896 30 August 1996 (30.08.96) US (71) Applicant (for all designated States except US): THE PROCTER & GAMBLE COMPANY [US/US]; One Procter & Gamble Plaza, Cincinnati, OH 45202 (US). (72) Inventors; and (75) Inventors/Applicants (for US only): WAHL, Errol, Hoffman [US/US]; 8021 Deersshadow Lane, Cincinnati, OH 45242 (US). TRINH, Toan [US/US]; 8671 Creekwood Lane, Maineville, OH 45039 (US). DEMEYERE, Hugo, Jean-Marie [BE/BE]; Linthout Straat 59, B-1785 Merchtem (BE). DECLERQ, Marc, Johan [BE/BE]; Ringlaan 77, B-1853 Strombeek-Bever (BE). (74) Agents: REED, T., David et al.; The Procter & Gamble Company, 5299 Spring Grove Avenue, Cincinnati, OH 45217 (US).		(81) Designated States: BR, CA, CN, JP, MX, US, European patent (AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE). Published <i>Without international search report and to be republished upon receipt of that report.</i>
(54) Title: CONCENTRATED PREMIX WITH REDUCED FLAMMABILITY FOR FORMING FABRIC SOFTENING COMPOSITION		
(57) Abstract Biodegradable fabric softener premix compositions are described containing (1) biodegradable softener actives; optional low molecular weight solvent; (2) high boiling, water soluble solvent to lower the viscosity, while avoiding flammability; and (3) optional perfume. The premixes can be added at ambient temperatures to water containing acid, to provide a pH of from about 1.5 to about 5, to form finished compositions. The water can also contain calcium and/or magnesium salt to modify the viscosity. Compositions containing highly unsaturated softener actives have improved freeze/thaw properties.		

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CONCENTRATED PREMIX WITH REDUCED FLAMMABILITY FOR
FORMING FABRIC SOFTENING COMPOSITION

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TECHNICAL FIELD

10 The present invention relates to concentrated liquid fabric softener active premixes for use in preparing softening compositions useful for softening cloth. It especially relates to the preparation of textile softening compositions for use in the rinse cycle of a home textile laundering operation to provide excellent fabric-softening/static-control benefits.

BACKGROUND OF THE INVENTION

15 Historically, fabric softening actives typically contain low solvent levels. However, normally, such fabric softener actives are difficult to process unless raised to high temperatures and processed with high energy levels. Raising the level of low molecular weight solvent to permit low temperature processing increases flammability. Moreover, there is a need for highly concentrated "compositions" that
20 are suitable either for sale to consumers as "finished compositions" or for creating such compositions. Said highly concentrated compositions that are used to further create finished compositions should have a viscosity that allows them to be stored, pumped, and processed at ambient temperatures into finished aqueous compositions without the need for high shear mixing. It is very desirable to have these
25 concentrated compositions also with low flammability or non-flammable characteristics. Concentrated active premixes and/or compositions that can be processed without difficulty, are of particular importance when one wants to provide the fabric softening benefit to consumers in remote areas of the world, where the cost of transporting finished low concentration products to the area from some other
30 location is prohibitively expensive and the local manufacturing facilities are limited and rudimentary. Safety and freeze/thaw stability are both important attributes for a consumer product and/or the products used to prepare such consumer products.

The present invention provides highly concentrated active compositions containing biodegradable actives with either no, or relatively low, levels of low
35 molecular weight organic solvent (e.g., below about 10%, by weight of the composition) so that they have reduced flammability, that can be processed at normal, i.e., room temperatures (about 25°C) and, preferably, even sub-normal

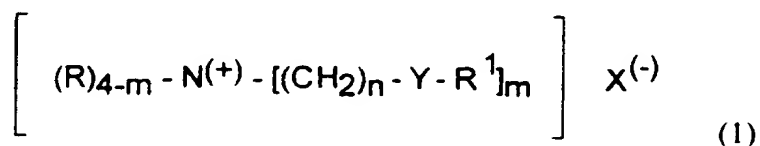
temperatures after prolonged storage conditions, preferably without high shear mixing, to form stable compositions.

SUMMARY OF THE INVENTION

The high active fabric softener "premix" compositions, useful for preparing finished compositions, herein comprise:

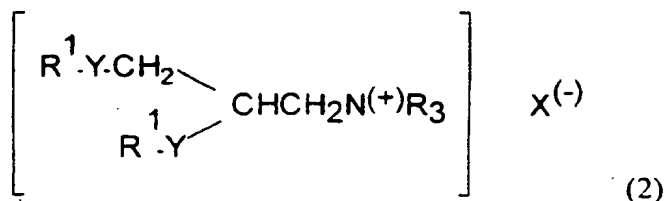
A. from about 50% to about 85%, preferably from about 60% to about 80%, more preferably from about 65% to about 75%, by weight of the composition, of biodegradable fabric softener active selected from the group consisting of:

1. softener having the formula:



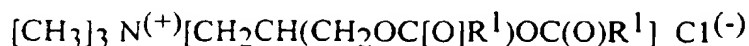
wherein each R substituent is a short chain C₁-C₆, preferably C₁-C₃ alkyl or hydroxyalkyl group, e.g., methyl (most preferred), ethyl, propyl, hydroxyethyl, and the like, benzyl, or mixtures thereof; each m is 2 or 3; each n is from 1 to about 4; each Y is -O-(O)C-, or -C(O)-O-; the sum of carbons in each R¹, plus one when Y is -O-(O)C-, is C₆-C₂₂, preferably C₁₄-C₂₀, and when one YR¹ sum is from about 6 to about 10, the other YR¹ sum is from about 16 to about 22, with each R¹ being hydrocarbyl, or substituted hydrocarbyl, substituent, preferably alkyl or alkylene; and where the total average Iodine Value (hereinafter referred to as IV) of the parent fatty acid of this R¹ group is preferably from about 40 to about 140, more preferably from about 60 to about 130; and most preferably from about 70 to about 105 (As used herein, the Iodine Value of the "parent" fatty acid, or "corresponding" fatty acid, is used to define an average level of unsaturation for all of the R¹ groups that are present, that is the same as the level of unsaturation that would be present in fatty acids containing the same R¹ groups.); and wherein the counterion, X⁻, can be any softener-compatible anion, preferably, chloride, methyl sulfate, bromide, or nitrate, more preferably chloride or methyl sulfate;

2. softener having the formula:



- 3 -

wherein each Y, R, R¹, and X⁽⁻⁾ have the same meanings as before (Such compounds include those having the formula:



- 5 where C(O)R¹ is derived from, e.g., natural source fatty acid including those that are high in oleic fatty acid, and, preferably, each R is a methyl or ethyl group and preferably each R¹ is in the range of C₁₅ to C₁₉ with degrees of branching and substitution optionally being present in the alkyl chains); and

3. mixtures thereof;

- 10 B. at least an effective amount to lower the viscosity, but less than about 40%, preferably from about 5% to about 30%, more preferably from about 7% to about 25%, and even more preferably from about 10% to about 20%, by weight, of high boiling point solvent, preferably to lower the viscosity to less than about 1000 cps, preferably less than about 500 cps, more preferably less than about 300 cps, while
- 15 not making the composition more flammable; and
- C optionally, from 0% to about 10%, preferably from about 3% to about 6%, by weight of the premix composition, of low molecular weight alcohol to lower the viscosity of the premix composition.

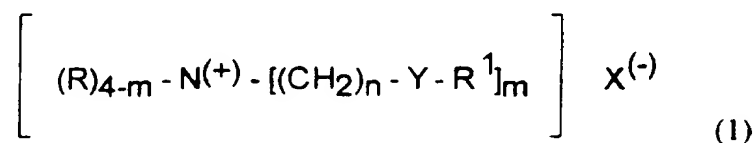
DETAILED DESCRIPTION OF THE INVENTION

20 A. FABRIC SOFTENING ACTIVE

The premixes herein contain, as an essential component, from about 50% to about 85%, preferably from about 60% to about 80%, more preferably from about 65% to about 75%, by weight of the composition, of a fabric softener active selected from the compounds identified hereinafter, and mixtures thereof.

25 Diester Quaternary Ammonium Fabric Softening Active Compound (DEQA)

(1) The first type of DEQA preferably comprises, as the principal active, compounds of the formula



30

wherein each R substituent is a short chain C₁-C₆, preferably C₁-C₃ alkyl or hydroxyalkyl group, e.g., methyl (most preferred), ethyl, propyl, hydroxyethyl, and the like, benzyl or mixtures thereof; each m is 2 or 3; each n is from 1 to about 4; each Y is -O-(O)C-, or -C(O)-O-; the sum of carbons in each R¹, plus one when Y is -O-(O)C-, is C₁₂-C₂₂, preferably C₁₄-C₂₀, with each R¹ being a hydrocarbyl, or

35

substituted hydrocarbyl, group, preferably alkyl or alkylene (mono- and/or poly-unsaturated), either straight and/or branched, and especially mixtures of groups derived from natural sources. Preferred compounds contain different R^1 groups. The average Iodine Value (hereinafter referred to as IV) of the parent fatty acid of this R^1 group is preferably from about 40 to about 140, more preferably from about 60 to about 130; and most preferably from about 70 to about 105.

The counterion, $X^{(-)}$ above, can be any softener-compatible anion, preferably the anion of a strong acid, for example, chloride, bromide, methylsulfate, sulfate, nitrate and the like, and more preferably chloride or methylsulfate.

These biodegradable quaternary ammonium fabric softening compounds can contain the group $C(O)R^1$ which is derived, primarily from unsaturated fatty acids, e.g., oleic acid, and/or fatty acids, and/or hydrogenated, and/or partially hydrogenated fatty acids, which are derived from vegetable oils, and/or partially hydrogenated vegetable oils, such as, canola oil, safflower oil, peanut oil, sunflower oil, corn oil, soybean oil, tall oil, rice bran oil, etc. Non-limiting examples of DEQAs prepared from fatty acids have the following approximate distributions:

Fatty Acyl

Group	DEQA ¹	DEQA ²	DEQA ³	DEQA ⁴	DEQA ⁵
C12	trace	trace	0	0	0
C14	3	3	0	0	0
C16	4	4	5	5	5
C18	0	0	5	6	6
C14:1	3	3	0	0	0
C16:1	11	7	0	0	3
C18:1	74	73	71	68	67
C18:2	4	8	8	11	11
C18:3	0	1	1	2	2
C20:1	0	0	2	2	2
C20 and up	0	0	2	0	0
Unknowns	0	0	6	6	7
Total	99	99	100	100	102
IV	86-90	88-95	99	100	95
cis/trans (C18:1)	20-30	20-30	4	5	5
TPU	4	9	11	13	13

Other nonlimiting examples of DEQA's of this invention are as follows:

Fatty Acyl Group	DEQA ⁶	DEQA ⁷
C14	0	1
C16	11	25

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C18	4	20
C14:1	0	0
C16:1	1	0
C18:1	27	45
C18:2	50	6
C18:3	7	0
Unknowns	0	3
<i>Total</i>	100	100

IV	125-138	56
cis/trans (C18:1)	Not Available	7
TPU	57	6

DEQA⁶ is prepared from a soy bean fatty acid, and DEQA⁷ is prepared from a slightly hydrogenated tallow fatty acid.

<u>Fatty Acyl Group</u>	<u>DEQA⁸</u>	<u>DEQA⁹</u>	<u>DEQA¹⁰</u>
Isomyristic acid	--	1-2	--
Myristic acid	7-11	0.5-1	--
Isopalmitic acid	6-7	6-7	1-3
Palmitic acid	4-5	6-7	--
Isostearic acid	70-76	80-82	60-66
Stearic acid	--	2-3	8-10
Isoleic acid	--	--	13-17
Oleic acid	--	--	6-12
IV	3	2	7-12

5

DEQA⁸ - DEQA¹⁰ are prepared from different commercially available isostearic acids.

Suitable DEQA's can be prepared as a single DEQA from blends of all the different fatty acids that are represented (total fatty acid blend), as well as from blends of mixtures of separate finished DEQA's that are prepared from different portions of the total fatty acid blend. Lower IV softener actives can be used if the branched chain fatty acids like isostearic acid are added.

The unsaturated, including the polyunsaturated, fatty acyl groups surprisingly provide effective softening, but also provide better rewetting characteristics, good antistatic characteristics, and especially, due to polyunsaturated fatty acids, superior recovery after freezing and thawing.

The highly unsaturated materials are also easier to formulate into concentrated premixes that maintain their low viscosity and are therefore easier to process, e.g., pump, mixing, etc. without large amounts of solvents. These highly unsaturated

materials with only the low amount of low molecular weight solvent that normally is associated with such materials, e.g., from about 7% to about 25%, preferably from about 10% to about 15%, weight of the total softener/solvent mixture, are already easier to formulate into concentrated, stable compositions of the present invention, even at ambient temperatures. However, at higher levels of such solvent, the concentrated premixes are flammable. The definition of flammable, is having a closed cup flash point of less than about 100 °F (about 38 °C). Higher boiling point, water soluble solvent, as described herein, can be used in preparation of the actives.

Lower IV actives (40-70) typically require larger amounts of higher boiling point water soluble solvent B.

This ability to safely process the actives at low temperatures is especially important for the polyunsaturated groups, since it minimizes degradation. Additional protection against degradation can be provided when the compounds and softener compositions contain effective antioxidants and/or reducing agents, as disclosed hereinafter.

It will be understood that substituents R and R¹ can optionally be substituted with various groups such as alkoxyl or hydroxyl groups, and can be straight, or branched so long as the R¹ groups maintain their basically hydrophobic character. Branched chains can be highly desirable, especially in mixtures of fatty acids, to lower the viscosity while still providing good stability. However, the present development allows the use of straight chain, saturated and unsaturated fatty acids, which are more readily available. The preferred compounds can be considered to be biodegradable diester variations of ditallow dimethyl ammonium chloride (hereinafter referred to as "DTDMAC"), which is a widely used fabric softener. Suitable long chain DEQAs are the DEQAs prepared from sources containing high levels of polyunsaturation, i.e., N,N-di(acyl-oxyethyl)-N,N-dimethyl ammonium chloride, where the acyl is derived from fatty acids containing sufficient polyunsaturation, e.g., mixtures of tallow fatty acids and soybean fatty acids.

If the fatty acyl groups are unsaturated, e.g., from about 50% to 100%, preferably from about 60% to about 98%, more preferably from about 65% to about 95%, and that the total level of polyunsaturated fatty acyl groups (TPU) be from about 1% to about 30%, preferably from about 3% to about 25%, more preferably from about 5% to about 18%, and even more preferably more than about 10%, then less solvent is used. The cis/trans ratio for the unsaturated fatty acyl groups is important, with the cis/trans ratio being from 1:1 to about 50:1, the minimum being 1:1, preferably at least 3:1, and more preferably from about 4:1 to about 30:1, again,

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to minimize use of solvent. Lower IV fatty acids can be used if blended with branched chain fatty acids, e.g., isostearic acid.

These biodegradable fabric softener actives typically contain a certain percentage, preferably low, e.g., less than about 5%, of free fatty acid. The free fatty acid also acts as a viscosity modifier, as disclosed hereinafter, and can add some softening benefit.

These premixes are desirable for formulating compositions with a lower concentration (from about 2% to about 50%, preferably from about 3% to about 30%, more preferably from about 4% to about 26%, by weight of the composition), stable compositions at ambient temperatures and/or without the use of high shear mixing, i.e., simple mixing is sufficient. It is understood, however, that high shear mixing and/or high temperatures can be used, if desired.

When the premixes are used to formulate higher concentration compositions, an additional advantage is that the concentrated compositions are not flammable. Finished compositions with concentrations of active of from about 13 to about 60, preferably from about 18 to about 52, can be created having viscosities of less than about 500cps, preferably less than about 400 cps, and more preferably less than about 200 cps, without danger of creating a fire hazard. Such compositions should have less than about 5%, preferably less than about 4%, more preferably less than about 3%, of low molecular weight solvent.

The preferred compounds can be considered to be biodegradable diester variations of ditallow dimethyl ammonium chloride (hereinafter referred to as "DTDMAC"), which is a widely used fabric softener. Preferably, at least about 80% of the DEQA is in the diester form, and from 0% to about 20% can be DEQA monoester (e.g., in formula (1), m is 2 and YR^1 is either H or a $-C(O)OH$ group). A highly preferred long chain DEQA is one containing a relatively high level of dioleoyl DEQA, i.e., N,N-di(oleoyl-oxyethyl)-N,N-dimethyl ammonium chloride. Preferred sources of fatty acids for use in preparing the DEQAs herein are the triglycerides, and/or partially hydrogenated triglycerides, from plants such as soybean oil, canola oil, safflower oil, peanut oil, sunflower oil, corn oil, soybean oil, tall oil, rice bran oil, etc. It is highly desirable that the compounds used herein are relatively free from unwanted impurities. Therefore, it is desirable to process the fatty acid sources in ways that are known to eliminate such impurities, e.g., processing under atmospheres that are low in oxygen, separating unwanted materials by filtration, adsorption, etc., either before and/or after chemical modification by controlled hydrogenation and/or oxygenation, etc. However, the purity of the materials is not part of the invention herein, which is equally applicable to less pure

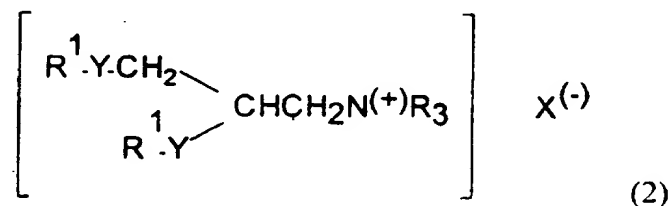
materials, the trade-off between purity and cost always being adjusted in light of the consumer's desires and needs.

As used herein, when the diester is specified, it can include the monoester that is present. For softening, under no/low detergent carry-over laundry conditions the percentage of monoester should be as low as possible, preferably no more than about 5%. However, under high, anionic detergent surfactant or detergent builder carry-over conditions, some monoester can be preferred. The overall ratios of diester to monoester are from about 100:1 to about 2:1, preferably from about 50:1 to about 5:1, more preferably from about 13:1 to about 8:1. Under high detergent carry-over conditions, the di/monoester ratio is preferably about 11:1. The level of monoester present can be controlled in manufacturing the DEQA.

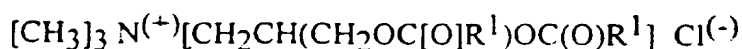
The above compounds, used as the biodegradable quaternized ester-amine softening material in the practice of this invention, can be prepared using standard reaction chemistry. In one synthesis of a di-ester variation of DTDMAC, an amine of the formula $RN(CH_2CH_2OH)_2$ is esterified at both hydroxyl groups with an acid chloride of the formula $R^1C(O)Cl$, then quaternized with an alkyl halide, RX , to yield the desired reaction product (wherein R and R^1 are as defined hereinbefore). However, it will be appreciated by those skilled in the chemical arts that this reaction sequence allows a broad selection of agents to be prepared.

Yet another DEQA softener active that is suitable for the formulation of the highly concentrated, liquid fabric softener premixes of the present invention, has the above formula (1) wherein one R group is a C_{1-4} hydroxyalkyl group, preferably one wherein one R group is a hydroxyethyl group. An example of such a hydroxyethyl ester active is di(acyloxyethyl)(2-hydroxyethyl)methyl ammonium methyl sulfate, where the acyl group is the same as that of DEQA¹.

(2) The second type of DEQA active has the general formula:



wherein each Y , R , R^1 , and $X^{(-)}$ have the same meanings as before. Such compounds include those having the formula:



where each R is a methyl or ethyl group and preferably each R¹ is in the range of C₁₅ to C₁₉. Degrees of branching and substitution can be present in the alkyl and/or alkylene chains. As used herein, when the diester is specified, it can include the monoester that is present. The amount of monoester that can be present is the same as in DEQA (1). An example of a DEQA of formula (2) is the "propyl" ester quaternary ammonium compound having the formula 1,2-di(acyloxy)-3-propanetrimethylammonium chloride, where the acyl group is the same as that for DEQA⁵.

These types of agents and general methods of making them are disclosed in U.S. Pat. No. 4,137,180, Naik et al., issued Jan. 30, 1979, which is incorporated herein by reference.

In softener actives (1) and (2), each R¹ is a hydrocarbyl, or substituted hydrocarbyl, group, preferably, alkyl, monounsaturated alkylene, and/or polyunsaturated alkylene groups. (As used herein, the "percent of softener active" containing a given R¹ group is the same as the percentage of that same R¹ group is to the total R¹ groups used to form all of the softener actives.) The actives preferably contain mixtures of R¹ groups, especially within the individual molecules, and also, optionally, the saturated R¹ groups can comprise branched chains, e.g., from isostearic acid, for at least part of the saturated R¹ groups (As used herein, the Iodine Value of a "parent" fatty acid, or "corresponding" fatty acid, is used to define a level of unsaturation for an R¹ group that is the same as the level of unsaturation that would be present in a fatty acid containing the same R¹ group.)

Compositions that can be prepared, include those described in the copending application of Errol H. Wahl et al. Serial No. 08/461,207, filed June 5, 1995 for "Concentrated Biodegradable Quaternary Ammonium Fabric Softener Compositions and Compounds Containing Intermediate Iodine Value Unsaturated Fatty Acid Chains," said application being incorporated herein by reference.

The DEQA actives described hereinbefore can contain a low level of free fatty acid which can be unreacted starting material and/or the by-product of a partial degradation(hydrolysis) of the softener active. It is preferred that the level of free fatty acid be low, preferably below about 10%, and more preferably below about 5%, by weight of the softener active.

B. HIGH BOILING WATER SOLUBLE ORGANIC SOLVENT

The premix compositions of the present invention comprise an effective amount to reduce viscosity up to about 40%, preferably from about 5% to about 30%, more preferably from about 7% to about 25%, and even more preferably from about 10% to about 20%, by weight of the premix composition of water soluble

organic solvent having a boiling point above about 85°C, more preferably above about 100°C. The flash point of the solvent should be higher than about 35°C, preferably higher than about 100°C, for safety reasons. These water soluble organic solvents are preferably totally miscible with water.

- 5 Non-limiting examples of the high boiling water soluble organic solvent includes diols (especially those described in the copending United States Patent Applications: 08/621,019; 08/620,627; 08/620,767; 08/620,513; 08/621,285; 08/621,299; 08/621,298; 08/620,626; 08/620,625; 08/620,772; 08/621,281; 08/620,514; and 08/620,958, all filed March 22, 1996 and all having the title
- 10 "CONCENTRATED, STABLE, PREFERABLY CLEAR, FABRIC SOFTENING COMPOSITION", all of said compositions being incorporated herein by reference), propylene carbonate, propylene glycol, hexylene glycol, butyl carbitol, and low molecular weight polyethylene glycols, although the polyethylene glycols are not preferred, and in some instances should be excluded to avoid problems in the
- 15 finished compositions, e.g., clear compositions as disclosed in said copending application. It is highly desirable to be able to formulate without the diols described in said patent applications, i.e., those solvents having a ClogP of from about 0.15 to about 0.64, preferably from about 0.25 to about 0.62, and more preferably from about 0.40 to about 0.60, and having a degree of asymmetry unless the eventual
- 20 finished composition is to be clear.

- Said high boiling water soluble organic solvent is selected, preferably, to provide the desired viscosity to the premix that will allow it to be processed at normal ambient temperatures. Such low viscosity and the ability to create finished compositions with only conventional mixing are more easily achieved with the
- 25 highly unsaturated fabric softener compounds disclosed hereinbefore. When solvent B., and any perfume that is present, as disclosed hereinafter, are insufficient to provide acceptable low viscosities for the premix, e.g., viscosities of from less than about 1000 cps, preferably less than about 500 cps, more preferably less than about 300 cps, at temperatures down to about 20°C, preferably down to about 10°C, then
- 30 alcohol C. as disclosed hereinafter, can be used to provide the needed viscosity reduction, and/or other desired properties, e.g., product clarity, so long as the desired low flammability is achieved.

- It is a surprising characteristic of these high boiling water soluble solvents that finished compositions containing them have superior freeze/thaw characteristics, if used with a high IV (IV >80) softener active. After they are
- 35 frozen, they recover more completely.

Preferred high boiling water soluble solvents for use herein are: 1,2-propylene glycol, hexylene glycol, butyl carbitol, etc.

C. **LOW MOLECULAR WEIGHT ALCOHOLS**

Low molecular weight alcohols, e.g., those with a molecular weight of less than about 120, especially less than about 100, are optionally present at levels of from 0% to about 10%, preferably from about 1% to about 7%, more preferably from about 3% to about 6%, by weight of the premix composition. These low molecular weight alcohols are normally inexpensive, usually come with the softener active, and lower the viscosity. Unfortunately, they are volatile and flammable. When the viscosity needs to be lowered, the level that can be used is limited, if one wishes to avoid having the product labeled "flammable" (closed cup flash point < 100 °F), thus creating the need for expensive shipping requirements. It has now been determined that one can use higher boiling solvents, to augment the low molecular weight solvents and improve the freeze-thaw recovery benefit, without causing problems in the finished products.

The level of such low molecular weight alcohols should be also be kept low for odor and VOC (volatile organic compounds) concerns for low molecular weight alcohols. For example, isopropyl alcohol and n-propyl alcohols have distinct odors and can create a fire hazard if used at high levels. However, these solvents can optionally be part of the total solvent present if levels are minimized. Several butyl alcohols also have odors but can be used for effective viscosity/stability, especially when used at low levels to minimize their odor. Such low molecular weight alcohols include: ethanol; isopropanol; n-propanol; t-butyl alcohol, etc. If the low molecular weight alcohols are insufficient, then one can add solvent B., as disclosed above.

D. **DISPERSIBILITY AIDS**

The premix of the present invention can optionally contain dispersibility aids, e.g., those selected from the group consisting of mono-long chain alkyl cationic quaternary ammonium compounds, mono-long chain alkyl amine oxides, ethoxylated fatty alcohols (C₁₂₋₂₂ alkyl/alkenyl, EO 5-40), ethoxylated fatty amines (C₁₂₋₂₂ alkyl/alkenyl, EO 5-40), other nonionic surfactants, and mixtures thereof, to assist in the formation of the finished compositions. These dispersibility aids are especially useful in the preparation of dilute softener compositions containing relatively high levels of perfume, as discussed hereinafter. When said dispersibility aid is present, it is typically present at a total level of from about 2% to about 35%, preferably from about 3% to about 25%, more preferably from about 4% to about 15%, and even more preferably from 5% to about 13% by weight of the

composition. These materials can either be added as part of the active softener raw material, (I), or added as a separate component in the preparation of the finished softener compositions. The total level of dispersibility aid includes any amount that may be present as part of component (I).

5 (1) Mono-Alkyl Cationic Quaternary Ammonium Compound

When the mono-alkyl cationic quaternary ammonium compound is present, it is typically present at a level of from about 2% to about 25%, preferably from about 3% to about 17%, more preferably from about 4% to about 15%, and even more preferably from 5% to about 13% by weight of the composition, the total
10 mono-alkyl cationic quaternary ammonium compound being at least at an effective level.

Such mono-alkyl cationic quaternary ammonium compounds useful in the present invention are, preferably, quaternary ammonium salts of the general formula:



wherein

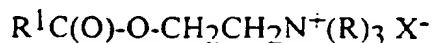
R^4 is C_8 - C_{22} alkyl or alkenyl group, preferably C_{10} - C_{18} alkyl or alkenyl group; more preferably C_{10} - C_{14} or C_{16} - C_{18} alkyl or alkenyl group;

each R^5 is a C_1 - C_6 alkyl or substituted alkyl group (e.g., hydroxy alkyl), preferably
20 C_1 - C_3 alkyl group, e.g., methyl (most preferred), ethyl, propyl, and the like, a benzyl group, hydrogen, a polyethoxylated chain with from about 2 to about 20 oxyethylene units, preferably from about 2.5 to about 13 oxyethylene units, more preferably from about 3 to about 10 oxyethylene units, and mixtures thereof; and
 X^- is as defined hereinbefore for (Formula (I)).

25 Especially preferred dispersibility aids are monolauryl trimethyl ammonium chloride and monotallow trimethyl ammonium chloride available from Witco under the trade name Varisoft® 471 and monooleyl trimethyl ammonium chloride available from Witco under the tradename Varisoft® 417.

The R^4 group can also be attached to the cationic nitrogen atom through a
30 group containing one, or more, ester, amide, ether, amine, etc., linking groups which can be desirable for increased concentratability of component (I), etc. Such linking groups are preferably within from about one to about three carbon atoms of the nitrogen atom.

Mono-alkyl cationic quaternary ammonium compounds also include C_8 - C_{22}
35 alkyl choline esters. The preferred dispersibility aids of this type have the formula:



wherein R^1 , R and X^- are as defined previously.

Highly preferred dispersibility aids include C₁₂-C₁₄ coco choline ester and C₁₆-C₁₈ tallow choline ester.

Suitable biodegradable single-long-chain alkyl dispersibility aids containing an ester linkage in the long chains are described in U.S. Pat. No. 4,840,738, Hardy and Walley, issued June 20, 1989, said patent being incorporated herein by reference.

When the dispersibility aid comprises alkyl choline esters, preferably the compositions also contain a small amount, preferably from about 2% to about 5% by weight of the composition, of organic acid. Organic acids are described in European Patent Application No. 404,471, Machin et al., published on Dec. 27, 1990, supra, which is herein incorporated by reference. Preferably the organic acid is selected from the group consisting of glycolic acid, acetic acid, citric acid, and mixtures thereof.

Ethoxylated quaternary ammonium compounds which can serve as the dispersibility aid include ethylbis(polyethoxy ethanol)alkylammonium ethyl-sulfate with 17 moles of ethylene oxide, available under the trade name Variquat® 66 from Witco Chemical Company; polyethylene glycol (15) oleammonium chloride, available under the trade name Ethoquad® 0/25 from Akzo Chemical Company; and polyethylene glycol (15) cocomonium chloride, available under the trade name Ethoquad® C/25 from Akzo Chemical Company.

Although the main function of the dispersibility aid is to increase the dispersibility of the ester softener, preferably the dispersibility aids of the present invention also have some softening properties to boost softening performance of the composition. Therefore, preferably the compositions of the present invention are essentially free of non-nitrogenous ethoxylated nonionic dispersibility aids which will decrease the overall softening performance of the compositions.

Also, quaternary compounds having only a single long alkyl chain, can protect the cationic softener from interacting with anionic surfactants and/or detergent builders that are carried over into the rinse from the wash solution.

(2) Amine Oxides

Suitable amine oxides include those with one alkyl or hydroxyalkyl moiety of about 8 to about 22 carbon atoms, preferably from about 10 to about 18 carbon atoms, more preferably from about 8 to about 14 carbon atoms, and two alkyl moieties selected from the group consisting of alkyl groups and hydroxyalkyl groups with about 1 to about 3 carbon atoms.

Examples include dimethyloctylamine oxide, diethyldecylamine oxide, bis-(2-hydroxyethyl)dodecyl-amine oxide, dimethyldodecylamine oxide, dipropyl-

tetradecylamine oxide, methylethylhexadecylamine oxide, dimethyl-2-hydroxyoctadecylamine oxide, and coconut fatty alkyl dimethylamine oxide.

Other nonionic surfactant dispersing aids useful in the present invention are disclosed in the copending provisional application of Cristina Avila-Garcia, et al.,
5 Serial No. 60/007,224, filed November 3, 1995, for "Stable High Perfume, Low-Active Fabric Softener Compositions", said application being incorporated herein by reference.

E. **OTHER OPTIONAL INGREDIENTS**

(A) **PERFUME**

10 The premixes and/or finished compositions of the present invention can contain any softener compatible perfume at a level that will provide the desired perfume effect in the final, finished product and lower the viscosity of the premix, thus improving processability. The level of the perfume is typically below about 25%, preferably from about 0.5% to about 15%, more preferably from about 1% to
15 about 10% to allow for dilution during the making of the final composition. The perfume should also be one, and at a level, that will not cause the product to be flammable. It is an advantage of the use of this invention, that the perfume, as well as the other optional solvents, and/or other ingredients, can be added in the premix to simplify the preparation of the finished compositions.

20 This convenient premix is possible, even with conventional actives. In conventional liquid fabric softener compositions, the softener actives are derived from fatty acids with lower IV values (less than about 60) and without this invention, they need to be heated and melted before they can be dispersed in water. Perfumes should not be exposed to the temperatures required for this melting. Some
25 perfume ingredients are degraded and some are volatilized and lost.

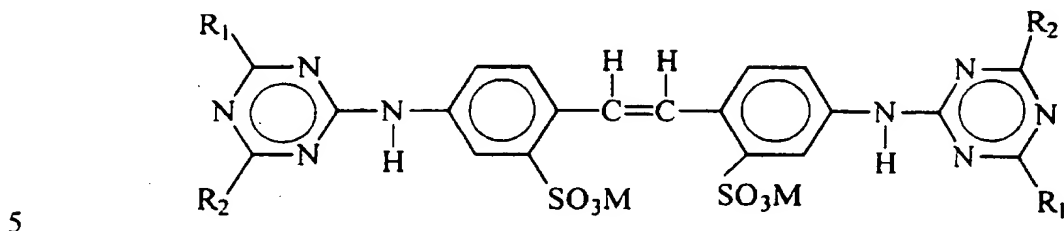
The premixes of the present invention, which contain perfume, can be added to water, desirably containing the requisite amount of acid, preferably mineral acid, more preferably HCl, at an ambient temperature, to create the finished composition as discussed hereinafter. Use of the premixes herein provides additional protection
30 for materials that can be degraded and avoids the use of high energy levels, thus lowering the total environmental load that is created in the manufacture of the finished compositions.

(B) **Brighteners**

The premix, and especially the finished compositions herein can also
35 optionally contain from about 0.005% to 5% by weight of certain types of hydrophilic optical brighteners which also provide a dye transfer inhibition action.

If used, the compositions herein will preferably comprise from about 0.001% to 1% by weight of such optical brighteners.

The hydrophilic optical brighteners useful in the present invention are those having the structural formula:



wherein R_1 is selected from anilino, N-2-bis-hydroxyethyl and NH-2-hydroxyethyl; R_2 is selected from N-2-bis-hydroxyethyl, N-2-hydroxyethyl-N-methylamino, morphilino, chloro and amino; and M is a salt-forming cation such as sodium or potassium.

10 When in the above formula, R_1 is anilino, R_2 is N-2-bis-hydroxyethyl and M is a cation such as sodium, the brightener is 4,4',-bis[(4-anilino-6-(N-2-bis-hydroxyethyl)-s-triazine-2-yl)amino]-2,2'-stilbenedisulfonic acid and disodium salt. This particular brightener species is commercially marketed under the tradename Tinopal-UNPA-GX[®] by Ciba-Geigy Corporation. Tinopal-UNPA-GX is the
15 preferred hydrophilic optical brightener useful in the rinse added compositions herein.

When in the above formula, R_1 is anilino, R_2 is N-2-hydroxyethyl-N-2-methylamino and M is a cation such as sodium, the brightener is 4,4'-bis[(4-anilino-6-(N-2-hydroxyethyl-N-methylamino)-s-triazine-2-yl)amino]-2,2'-stilbenedisulfonic
20 acid disodium salt. This particular brightener species is commercially marketed under the tradename Tinopal 5BM-GX[®] by Ciba-Geigy Corporation.

When in the above formula, R_1 is anilino, R_2 is morphilino and M is a cation such as sodium, the brightener is 4,4'-bis[(4-anilino-6-morphilino-s-triazine-2-yl)amino]-2,2'-stilbenedisulfonic acid, sodium salt. This particular brightener species
25 is commercially marketed under the tradename Tinopal AMS-GX[®] by Ciba Geigy Corporation.

(C) Stabilizers

Stabilizers can be present in the premixes and finished compositions of the present invention. The term "stabilizer," as used herein, includes antioxidants and
30 reductive agents. These agents are present at a level of from 0% to about 2%, preferably from about 0.01% to about 0.2%, more preferably from about 0.035% to about 0.1% for antioxidants, and more preferably from about 0.01% to about 0.2% for reductive agents, in the final composition. For the premix, the levels are

- 16 -

adjusted, depending on the concentrations of the softener active in the premix and the finished composition. These assure good odor stability under long term storage conditions. Antioxidants and reductive agent stabilizers are especially critical for unscented or low scent products (no or low perfume).

5 Examples of antioxidants that can be added to the compositions of this invention include a mixture of ascorbic acid, ascorbic palmitate, propyl gallate, available from Eastman Chemical Products, Inc., under the trade names Tenox® PG and Tenox® S-1; a mixture of BHT (butylated hydroxytoluene), BHA (butylated hydroxyanisole), propyl gallate, and citric acid, available from Eastman Chemical
10 Products, Inc., under the trade name Tenox®-6; butylated hydroxytoluene, available from UOP Process Division under the trade name Sustane® BHT; tertiary butylhydroquinone, Eastman Chemical Products, Inc., as Tenox® TBHQ; natural tocopherols, Eastman Chemical Products, Inc., as Tenox® GT-1/GT-2; and butylated hydroxyanisole, Eastman Chemical Products, Inc., as BHA; long chain
15 esters (C₈-C₂₂) of gallic acid, e.g., dodecyl gallate; Irganox® 1010; Irganox® 1035; Irganox® B 1171; Irganox® 1425; Irganox® 3114; Irganox® 3125; and mixtures thereof; preferably Irganox® 3125, Irganox® 1425, Irganox® 3114, and mixtures thereof; more preferably Irganox® 3125 alone or mixed with citric acid and/or other chelators such as isopropyl citrate, Dequest® 2010, available from Monsanto with a
20 chemical name of 1-hydroxyethylidene-1, 1-diphosphonic acid (etidronic acid), and Tiron®, available from Kodak with a chemical name of 4,5-dihydroxy-m-benzene-sulfonic acid/sodium salt, and DTPA®, available from Aldrich with a chemical name of diethylenetriaminepentaacetic acid.

(D) Soil Release Agent

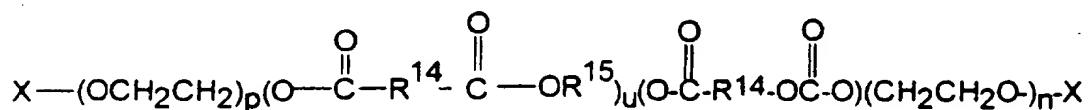
25 In the present invention, an optional soil release agent can be added. The addition of the soil release agent can occur in combination with the premix, in combination with the acid/water seat, before or after electrolyte addition, or after the final composition is made. The finished softening composition prepared by the process of the present invention herein can contain from 0% to about 10%,
30 preferably from 0.2% to about 5%, of a soil release agent. The concentration in the premix is adjusted to provide the desired end concentration. Preferably, such a soil release agent is a polymer. Polymeric soil release agents useful in the present invention include copolymeric blocks of terephthalate and polyethylene oxide or polypropylene oxide, and the like.

35 A preferred soil release agent is a copolymer having blocks of terephthalate and polyethylene oxide. More specifically, these polymers are comprised of repeating units of ethylene terephthalate and polyethylene oxide terephthalate at a

molar ratio of ethylene terephthalate units to polyethylene oxide terephthalate units of from 25:75 to about 35:65, said polyethylene oxide terephthalate containing polyethylene oxide blocks having molecular weights of from about 300 to about 2000. The molecular weight of this polymeric soil release agent is in the range of
 5 from about 5,000 to about 55,000.

Another preferred polymeric soil release agent is a crystallizable polyester with repeat units of ethylene terephthalate units containing from about 10% to about 15% by weight of ethylene terephthalate units together with from about 10% to about 50% by weight of polyoxyethylene terephthalate units, derived from a
 10 polyoxyethylene glycol of average molecular weight of from about 300 to about 6,000, and the molar ratio of ethylene terephthalate units to polyoxyethylene terephthalate units in the crystallizable polymeric compound is between 2:1 and 6:1. Examples of this polymer include the commercially available materials Zelcon 4780[®] (from Dupont) and Milease T[®] (from ICI).

15 Highly preferred soil release agents are polymers of the generic formula:



in which each X can be a suitable capping group, with each X typically being
 20 selected from the group consisting of H, and alkyl or acyl groups containing from about 1 to about 4 carbon atoms. p is selected for water solubility and generally is from about 6 to about 113, preferably from about 20 to about 50. u is critical to formulation in a liquid composition having a relatively high ionic strength. There should be very little material in which u is greater than 10. Furthermore, there
 25 should be at least 20%, preferably at least 40%, of material in which u ranges from about 3 to about 5.

The R¹⁴ moieties are essentially 1,4-phenylene moieties. As used herein, the term "the R¹⁴ moieties are essentially 1,4-phenylene moieties" refers to compounds where the R¹⁴ moieties consist entirely of 1,4-phenylene moieties, or
 30 are partially substituted with other arylene or alkarylene moieties, alkylene moieties, alkenylene moieties, or mixtures thereof. Arylene and alkarylene moieties which can be partially substituted for 1,4-phenylene include 1,3-phenylene, 1,2-phenylene, 1,8-naphthylene, 1,4-naphthylene, 2,2-biphenylene, 4,4-biphenylene, and mixtures thereof. Alkylene and alkenylene moieties which can be partially substituted
 35 include 1,2-propylene, 1,4-butylene, 1,5-pentylene, 1,6-hexamethylene, 1,7-heptamethylene, 1,8-octamethylene, 1,4-cyclohexylene, and mixtures thereof.

For the R^{14} moieties, the degree of partial substitution with moieties other than 1,4-phenylene should be such that the soil release properties of the compound are not adversely affected to any great extent. Generally the degree of partial substitution which can be tolerated will depend upon the backbone length of the compound, i.e., longer backbones can have greater partial substitution for 1,4-phenylene moieties. Usually, compounds where the R^{14} comprise from about 50% to about 100% 1,4-phenylene moieties (from 0% to about 50% moieties other than 1,4-phenylene) have adequate soil release activity. For example, polyesters made according to the present invention with a 40:60 mole ratio of isophthalic (1,3-phenylene) to terephthalic (1,4-phenylene) acid have adequate soil release activity. However, because most polyesters used in fiber making comprise ethylene terephthalate units, it is usually desirable to minimize the degree of partial substitution with moieties other than 1,4-phenylene for best soil release activity. Preferably, the R^{14} moieties consist entirely of (i.e., comprise 100%) 1,4-phenylene moieties, i.e., each R^{14} moiety is 1,4-phenylene.

For the R^{15} moieties, suitable ethylene or substituted ethylene moieties include ethylene, 1,2-propylene, 1,2-butylene, 1,2-hexylene, 3-methoxy-1,2-propylene, and mixtures thereof. Preferably, the R^{15} moieties are essentially ethylene moieties, 1,2-propylene moieties, or mixtures thereof. Inclusion of a greater percentage of ethylene moieties tends to improve the soil release activity of compounds. Surprisingly, inclusion of a greater percentage of 1,2-propylene moieties tends to improve the water solubility of compounds.

Therefore, the use of 1,2-propylene moieties or a similar branched equivalent is desirable for incorporation of any substantial part of the soil release component in the liquid fabric softener compositions. Preferably, from about 75% to about 100%, are 1,2-propylene moieties.

The value for each p is at least about 6, and preferably is at least about 10. The value for each n usually ranges from about 12 to about 113. Typically the value for each p is in the range of from about 12 to about 43.

A more complete disclosure of soil release agents is contained in U.S. Pat. Nos.: 4,661,267, Decker, Konig, Straathof, and Gosselink, issued Apr. 28, 1987; 4,711,730, Gosselink and Diehl, issued Dec. 8, 1987; 4,749,596, Evans, Huntington, Stewart, Wolf, and Zimmerer, issued June 7, 1988; 4,818,569, Trinh, Gosselink, and Rattinger, issued April 4, 1989; 4,877,896, Maldonado, Trinh, and Gosselink, issued Oct. 31, 1989; 4,956,447, Gosselink et al., issued Sept. 11, 1990; and 4,976,879, Maldonado, Trinh, and Gosselink, issued Dec. 11, 1990, all of said patents being incorporated herein by reference.

These soil release agents can also act as scum dispersants.

(E) Scum Dispersant

In the present invention, the premix can be combined with an optional scum dispersant, other than the soil release agent, and heated to a temperature at or above the melting point(s) of the components.

The preferred scum dispersants herein are highly ethoxylated hydrophobic materials. The hydrophobic material can be a fatty alcohol, fatty acid, fatty amine, fatty acid amide, amine oxide, quaternary ammonium compound, or the hydrophobic moieties used to form soil release polymers. The preferred scum dispersants are highly ethoxylated, e.g., more than about 17, preferably more than about 25, more preferably more than about 40, moles of ethylene oxide per molecule on the average, with the polyethylene oxide portion being from about 76% to about 97%, preferably from about 81% to about 94%, of the total molecular weight.

The level of scum dispersant is sufficient to keep the scum at an acceptable, preferably unnoticeable to the consumer, level under the conditions of use, but not enough to adversely affect softening. For some purposes it is desirable that the scum is nonexistent. Depending on the amount of anionic or nonionic detergent, etc., used in the wash cycle of a typical laundering process, the efficiency of the rinsing steps prior to the introduction of the compositions herein, and the water hardness, the amount of anionic or nonionic detergent surfactant and detergency builder (especially phosphates and zeolites) entrapped in the fabric (laundry) will vary. Normally, the minimum amount of scum dispersant should be used to avoid adversely affecting softening properties. Typically scum dispersion requires at least about 2%, preferably at least about 4% (at least 6% and preferably at least 10% for maximum scum avoidance) based upon the level of softener active. However, at levels of about 10% (relative to the softener material) or more, one risks loss of softening efficacy of the product especially when the fabrics contain high proportions of nonionic surfactant which has been absorbed during the washing operation.

Preferred scum dispersants are: Brij 700®; Varonic U-250®; Genapol T-500®, Genapol T-800®; Plurafac A-79®; and Neodol 25-50®.

(F) Bactericides

Examples of bactericides which can be present in the premixes and finished compositions of this invention include glutaraldehyde, formaldehyde, 2-bromo-2-nitro-propane-1,3-diol sold by Inolex Chemicals, located in Philadelphia, Pennsylvania, under the trade name Bronopol®, and a mixture of 5-chloro-2-methyl-4-isothiazoline-3-one and 2-methyl-4-isothiazoline-3-one sold by Rohm and

Haas Company under the trade name Kathon CG/ICP®. Typical levels of bactericides used in the present compositions are from about 1 to about 1,000 ppm by weight of the agent.

(G) Chelating Agents

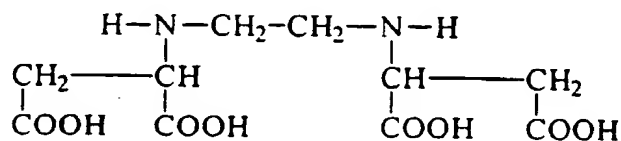
5 The compositions and processes herein can optionally employ one or more copper and/or nickel chelating agents ("chelators"). Such water-soluble chelating agents can be selected from the group consisting of amino carboxylates, amino phosphonates, polyfunctionally-substituted aromatic chelating agents and mixtures thereof, all as hereinafter defined. The whiteness and/or brightness of fabrics are
10 substantially improved or restored by such chelating agents and the stability of the materials in the compositions are improved.

Amino carboxylates useful as chelating agents herein include ethylenediaminetetraacetates (EDTA), N-hydroxyethylethylenediaminetriacetates, nitrilotriacetates (NTA), ethylenediamine tetrapropionates, ethylenediamine-N,N'-
15 diglutamates, 2-hydroxypropylenediamine-N,N'-disuccinates, triethylenetetraaminehexacetates, diethylenetriaminepentaacetates (DETPA), and ethanoldiglycines, including their water-soluble salts such as the alkali metal, ammonium, and substituted ammonium salts thereof and mixtures thereof.

Amino phosphonates are also suitable for use as chelating agents in the
20 compositions of the invention when at least low levels of total phosphorus are permitted in detergent compositions, and include ethylenediaminetetrakis (methylenephosphonates), diethylenetriamine-N,N,N',N'',N''-pentakis(methane phosphonate) (DETMP) and 1-hydroxyethane-1,1-diphosphonate (HEDP). Preferably, these amino phosphonates to not contain alkyl or alkenyl groups with
25 more than about 6 carbon atoms.

The chelating agents are typically used in the present rinse process at levels from about 2 ppm to about 25 ppm, for periods from 1 minute up to several hours' soaking.

The preferred EDDS chelator used herein (also known as ethylenediamine-N,N'-disuccinate) is the material described in U.S. Patent 4,704,233, cited
30 hereinabove, and has the formula (shown in free acid form):



As disclosed in the patent, EDDS can be prepared using maleic anhydride and ethylenediamine. The preferred biodegradable [S,S] isomer of EDDS can be
35 prepared by reacting L-aspartic acid with 1,2-dibromoethane. The EDDS has

advantages over other chelators in that it is effective for chelating both copper and nickel cations, is available in a biodegradable form, and does not contain phosphorus. The EDDS employed herein as a chelator is typically in its salt form, i.e., wherein one or more of the four acidic hydrogens are replaced by a water-soluble cation M, such as sodium, potassium, ammonium, triethanolammonium, and the like. As noted before, the EDDS chelator is also typically used in the present rinse process at levels from about 2 ppm to about 25 ppm for periods from 1 minute up to several hours' soaking. At certain pH's the EDDS is preferably used in combination with zinc cations.

As can be seen from the foregoing, a wide variety of chelators can be used herein. Indeed, simple polycarboxylates such as citrate, oxydisuccinate, and the like, can also be used, although such chelators are not as effective as the amino carboxylates and phosphonates, on a weight basis. Accordingly, usage levels may be adjusted to take into account differing degrees of chelating effectiveness. The chelators herein will preferably have a stability constant (of the fully ionized chelator) for copper ions of at least about 5, preferably at least about 7. Typically, the chelators will comprise from about 0.5% to about 10%, more preferably from about 0.75% to about 5%, by weight of the compositions herein. Preferred chelators include DETMP, DETPA, NTA, EDDS and mixtures thereof.

(H) Optional Viscosity/Dispersibility Modifiers

Relatively concentrated compositions containing both saturated and unsaturated diester quaternary ammonium compounds can be prepared that are stable without the addition of concentration aids. However, the compositions of the present invention may require organic and/or inorganic concentration aids to go to even higher concentrations and/or to meet higher stability standards depending on the other ingredients. These concentration aids which typically can be viscosity modifiers may be needed, or preferred, for ensuring stability under extreme conditions when particular softener active levels are used. The surfactant concentration aids are typically selected from the group consisting of (1) single long chain alkyl cationic surfactants; (2) nonionic surfactants; (3) amine oxides; (4) fatty acids; and (5) mixtures thereof. These aids are described in P&G Copending Application Serial No. 08/461,207, filed June 5, 1995, Wahl et al., specifically on page 14, line 12 to page 20, line 12, which is herein incorporated by reference. Fatty acids containing from about 12 to about 18 carbon atoms, especially from about 16 to about 18 carbon atoms, are desirable additives for this purpose, since they can provide additional softening.

(I) Other Optional Ingredients

The present invention can include optional components conventionally used in textile treatment compositions, for example: colorants; preservatives; surfactants; anti-shrinkage agents; fabric crisping agents; spotting agents; germicides; fungicides; anti-corrosion agents; and the like.

The present invention can also include other compatible ingredients, including those as disclosed in copending applications Serial Nos.: 08/372,068, filed January 12, 1995, Rusche, et al.; 08/372,490, filed January 12, 1995, Shaw, et al.; and 08/277,558, filed July 19, 1994, Hartman, et al., incorporated herein by reference.

PROCESSING ASPECTS

The premixes comprise the softener active A. (from about 50% to about 85%, preferably from about 60% to about 80%, more preferably from about 65% to about 75%, by weight of the premix); the higher boiling water soluble organic solvent system B. at the levels specified hereinbefore; optionally, the low molecular weight alcohol C. (from 0% to about 10%, preferably from about 1% to about 7%, more preferably from about 3% to about 5%, by weight of the premix (Preferably the level of low molecular weight alcohol is sufficiently low so that the closed cup flammability point is greater than about 100 °F.); and optionally, the perfume (up to about 50% for the high perfume, low active compositions described hereinafter, and less than about 25%, preferably from about 0.5% to about 15%, more preferably from about 1% to about 10% by weight of the premix, for more normal, high softener concentration compositions); and optionally, the dispersibility aid E. (from 0% to about 35%, preferably from about 3% to about 25%, and more preferably from about 4% up to about 15%, by weight of the premix). The higher boiling solvent reduces the level of low molecular weight solvent required to achieve the desired viscosities while reducing, or eliminating, flammability.

The development especially relates to compositions wherein the active and the low molecular weight solvent mixture does not have a viscosity that permits facile processing. The development includes the process of adding the high boiling water soluble solvent to the composition to lower the viscosity and make it processable, while keeping it non-flammable, or of low flammability. This same benefit is also observed in finished compositions which contain a high level of active and which need to have the viscosity reduced. In both premixes and high active finished compositions, the high boiling water soluble solvent provides a low viscosity and reduced flammability.

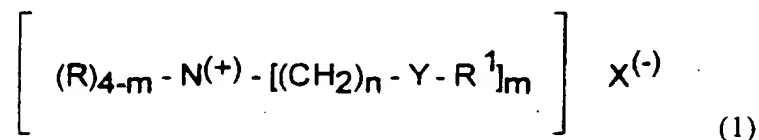
The compositions prepared using these premixes, and/or compositions containing said higher boiling, water soluble solvent, have improved freeze/thaw properties, (recovery after freezing) if a high IV (>80) softener active is used. This property is totally unexpected.

- 5 These premixes have, typical viscosities suitable for processing of less than about 1000 cps, preferably less than about 500 cps, more preferably less than about 300 cps. The finished compositions can be made by adding a preheated softener premix to a preheated water seat, but are preferably made at ambient temperatures. Use of low temperatures improves safety, by minimizing solvent vaporization,
 10 minimizes the degradation and/or loss of materials such as the biodegradable fabric softener active, perfumes, preservatives, etc., and reduces the need for heating, thus saving on the expenses for processing. The result is improved environmental impact and safety from the manufacturing operation.

- Examples of premixes and processes using them include premixes which
 15 typically contain from about 50% to about 85%, preferably from about 60% to about 80%, more preferably from about 65% to about 75%, of fabric softener active A.; up to about 40% of the high boiling point water soluble solvent B.; and from about 1% to about 7%, preferably from about 3% to about 5%, of low molecular weight water soluble solvent C., like ethanol and/or isopropanol, or the preferred levels set forth
 20 hereinbefore.

 Some preferred finished fabric softener compositions that can be prepared using the premixes herein comprise:

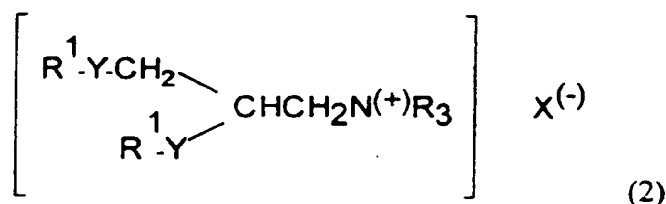
- A. from about 5% to about 50%, preferably from about 10% to about 35%, more preferably from about 17% to about 30%, by weight of the composition, of
 25 biodegradable fabric softener active selected from the group consisting of:
 1. softener having the formula:



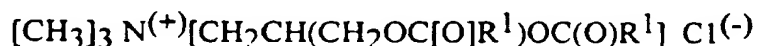
- 30 wherein each R substituent is a short chain C₁-C₆, preferably C₁-C₃ alkyl or hydroxyalkyl group, e.g., methyl (most preferred), ethyl, propyl, hydroxyethyl, and the like, benzyl, or mixtures thereof; each m is 2 or 3; each n is from 1 to about 4; each Y is -O-(O)C-, or -C(O)-O-; the sum of carbons in each R¹, plus one when Y is -O-(O)C-, is C₁₂-C₂₂, preferably C₁₄-C₂₀, with each R¹ being a hydrocarbyl, or
 35 substituted hydrocarbyl group, preferably, alkyl, monounsaturated alkylene, and

polyunsaturated alkylene groups (As used herein, the "percent of softener active" containing a given R^1 group is based upon taking a percentage of the total active based upon the percentage that the given R^1 group is, of the total R^1 groups present and the Iodine Value of a "parent" fatty acid, or "corresponding" fatty acid, is used to define a level of unsaturation for an R^1 group that is the same as the level of unsaturation that would be present in a fatty acid containing the same R^1 group.); and wherein the counterion, X^- , can be any softener-compatible anion, preferably, chloride, bromide, or nitrate, more preferably chloride;

2. softener having the formula:



wherein each Y, R, R^1 , and $X^{(-)}$ have the same meanings as before (Such compounds include those having the formula:



especially where $C(O)R^1$ is derived from mixtures of R^1 groups, containing some saturated, some unsaturated, e.g., oleic, fatty acid, and, preferably, each R is a methyl or ethyl group and preferably each R^1 is in the range of C_{15} to C_{19} with degrees of branching and unsaturation being present in the alkyl chains); and

3. mixtures thereof; said fabric softener active being in the form of a stable dispersion; and

B. the balance water and from about 1% to about 30%, preferably from about 3% to about 25%, more preferably from about 8% to about 20%, by weight of the composition of water soluble organic solvent, the level of low molecular weight organic solvent being too low to cause the composition to be flammable; the viscosity of the composition being less than about 500 cps, preferably less than about 400 cps, more preferably less than about 200 cps, and recovering to less than about 1000 cps, preferably less than about 500 cps, more preferably less than about 200 cps after freezing and thawing.

Particularly preferred optional ingredients for use in the compositions of this type, include water soluble, ionizable, calcium and/or magnesium salts, which provide desired viscosity and/or additional stability. The chloride salts are

preferred, but acetate, nitrate, etc. salts can be used. The level of said calcium and/or magnesium salts is from 0% to about 2%, preferably from about 0.05% to about 0.5%, more preferably from about 0.1% to about 0.25%. Sodium salts such as NaCl can also be used, especially for compositions containing low levels of softener actives. These materials are desirably in the water and/or acid (water seat) used to prepare the finished compositions. The pH of the finished compositions should be from about 1 to about 7, preferably from about 1.5 to about 5, more preferably from about 2 to about 3.5. The pH is normally adjusted by using a low molecular weight acid, preferably mineral acid, more preferably HCl. Other acids, including nitric acid, acetic acid, etc., can be used to provide the pH.

Other compositions that can be prepared using the premixes herein include the "clear" compositions described in the copending United States Patent Applications: 08/621,019; 08/620,627; 08/620,767; 08/620,513; 08/621,285; 08/621,299; 08/621,298; 08/620,626; 08/620,625; 08/620,772; 08/621,281; 08/620,514; and 08/620,958, all filed March 22, 1996 and all having the title "CONCENTRATED, STABLE, PREFERABLY CLEAR, FABRIC SOFTENING COMPOSITION", all of said compositions being incorporated herein by reference.

Other low softener, high perfume, compositions, disclosed in the copending provisional application of Cristina Avila-Garcia, et al., Serial No. 60/007,224, filed November 3, 1995, for "Stable High Perfume, Low-Active Fabric Softener Compositions", said application being incorporated hereinbefore by reference, can be prepared using the premixes including: single strength liquid fabric softener compositions for use in the rinse cycle of a laundering process, the compositions comprising:

- (a) from about 0.4% to about 5% cationic fabric softener;
- (b) from about 0.3% to about 1.2% hydrophobic perfume;
- (c) from about 0.4% to about 5% nonionic surfactant dispersibility aid;
- (d) from 0% to about 1% water-soluble ionizable inorganic salt;
- (e) from about 90% to about 98.5% water;
- (f) an effective amount up to about 40%, of high boiling water soluble solvent; and
- (g) from 0% to about 2% other ingredients;

the ratio of cationic softener to perfume being from about 1:3 to about 5:1; the ratio of cationic softener to nonionic surfactant being from about 1:2 to about 4:1, and the amount of cationic softener plus nonionic surfactant being from about 1% to about 7%. The compositions consist of a liquid aqueous phase with discrete hydrophobic

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particles dispersed substantially uniformly therein. The compositions preferably have a viscosity of from about 50 cp to about 500 cp.

The premixes can be used to formulate finished compositions in simple processes, at ambient temperatures, comprising the steps of:

1. Make up water seat of water and HCl at ambient temperature. Optionally add chelant.
2. Add premix, eg., as disclosed below, to water under good agitation.
3. Trim with CaCl_2 solution to desired viscosity.
4. Add dye solution to get desired color and add any other minors.
5. Add perfume if it is not in the premix.

For improved low temperature viscosity stability at high concentrations of active, the premix and water seat should be heated during mixing.

The fabric softening actives (DEQAs); the high boiling, water soluble solvent; and, optionally, the low molecular weight solvent, can be formulated as premixes as follows.

PREMIXES

Component (Wt%)	I	II	III	IV	V
DEQA ⁵	72.3	72.3	72.3	72.3	72.3
Ethanol	6.35	6.35	--	6.35	--
Hexylene Glycol	6.35	21.35	27.7	6.35	12.7
Propylene Glycol	15.0	--	--	--	15.0
Butyl Carbitol	--	--	--	15.0	--

These premixes can produce clear or opaque fabric softeners compositions that are non-flammable (i.e., have a closed cup flash point of >100F). The fabric softener compositions below (Compositions 1-5) can be prepared using Premix I.

Compositions 1-5

Composition	1	2	3	4	5
Formula #	26% Active	8.5% Active	10.5% Active	Concentrated	High Perfume
Component:	(%)	(%)	(%)	(%)	(%)
DEQA ⁵ (100%)	26.0	8.50	10.50	42.5	8.5
EtOH	2.3	0.75	0.92	3.7	0.75
Hexylene Glycol	2.3	0.75	0.92	3.7	0.75
Propylene Glycol	5.4	1.76	2.18	8.8	1.76
TMPD [*]	15.0	-	-	22.0	-
1,4-CHDM ^{**}	5.0	-	-	8.0	-
HCl (1N)	0.25	-	-	0.4	-

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HCl (32%)	-	0.06	0.06	-	0.06
Silicone emulsion	-	0.005	0.005	-	0.01
Calcium Chloride	-	0.15	0.015	-	0.029
Dye	0.0003	0.001	0.001	-	0.005
Soil Release Polymer	-	-	-	-	0.19
PEG4000 (Polyethylene glycol)	-	-	-	-	0.6
EDDS	-	0.025	0.025	-	0.025
Kathon	0.0003	-	-	0.0003	-
DTPA	0.01	-	-	0.01	-
Gluteraldehyde	-	0.025	0.025	-	0.025
Perfume	1.25	0.40	1.75	1.25	1.75
DI Water	42.49	87.57	83.46	9.64	85.55

* 2,2,4-trimethyl-1,3-pentanediol

** 1,4-cyclohexanedimethanol

The fabric softener compositions below (Compositions 6-9) can be prepared
5 using Premix V.

Compositions 6-9

Composition	1	2	3	4	5
Formula #	26% Active	8.5% Active	10.5% Active	Concentrated	High Perfume
Component:	(%)	(%)	(%)	(%)	(%)
DEQA ^S (100%)	26.00	8.50	10.50	42.5	8.5
EtOH	2.3	0.75	0.92	3.7	0.75
Hexylene Glycol	2.3	0.75	0.92	3.7	0.75
Propylenglycol	5.4	1.76	2.18	8.8	1.76
TMPD*	15.00	-	-	22.0	-
1,4-CHDM**	5.00	-	-	8.0	-
HCl (1N)	0.25	-	-	0.4	-
HCl (32%)	-	0.06	0.06	-	0.06
Silicone emulsion	-	0.005	0.005	-	0.01
Calcium Chloride	-	0.15	0.15	-	0.029
Dye	0.0003	0.001	0.001	-	0.005
Soil Release Polymer	-	-	-	-	0.19
PEG4000 (Polyethylene glycol)	-	-	-	-	0.6
EDDS	-	0.025	0.025	-	0.025
Kathon	0.0003	-	-	0.0003	-
DTPA	0.01	-	-	-	-
Gluteraldehyde	-	0.025	0.025	-	0.025
Perfume	1.25	0.40	1.75	1.25	1.75
DI Water	48.09	87.57	83.46	9.64	85.55

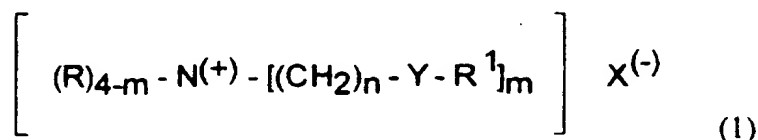
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WHAT IS CLAIMED IS:

1. A fabric softener premix composition, useful for preparing finished compositions, comprising:

A. from about 50% to about 85% of fabric softener active selected from the group consisting of:

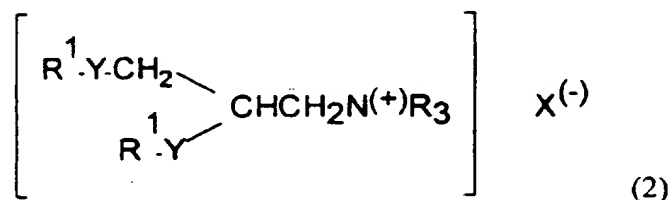
5 1. fabric softener compound having the formula:



10 wherein each R substituent is a short chain C₁-C₆ alkyl or hydroxyalkyl group, benzyl, or mixtures thereof; each m is 2 or 3; each n is from 1 to about 4; each Y is -O-(O)C-, or -C(O)-O-; the sum of carbons in each R¹, plus one when Y is -O-(O)C-, is C₁₂-C₂₂, with R¹ being hydrocarbyl, or substituted hydrocarbyl, substituent; and the Iodine Value of the fatty acid which contains this

15 R¹ group is from about 40 to about 140;

2. fabric softener compound having the formula:



20 wherein each Y, R, R¹, and X⁽⁻⁾ have the same meanings as before; and

3. mixtures thereof;

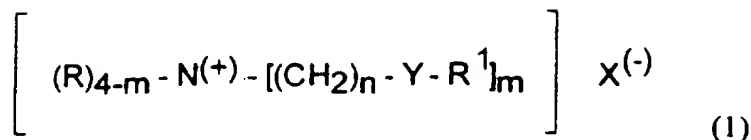
25 B. from an effective amount to about 40% by weight of the premix composition of high boiling water soluble organic solvent to reduce the viscosity; and

C. optionally, an effective amount, sufficient to lower the viscosity, of low molecular weight water soluble solvents.

2. The fabric softener premix composition of Claim 1 comprising:

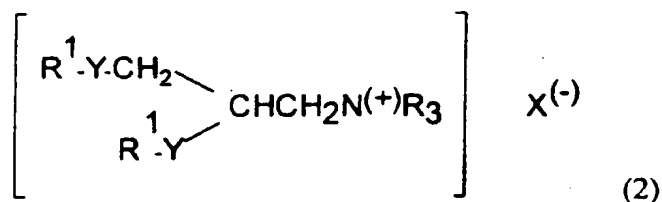
A. from about 60% to about 80% of said fabric softener active selected from the group consisting of:

1. fabric softener compound having the formula:



wherein each R substituent is a short chain C₁-C₃ alkyl or hydroxyalkyl group, benzyl or mixtures thereof; each m is 2; each n is from 2 to about 3; each Y is -O-(O)C-; the sum of carbons in each R¹ plus one is C₁₄-C₂₀; the Iodine Value of the corresponding fatty acid of this R¹ group is from about 60 to about 130,;

2. fabric softener compound having the formula:



wherein each Y, R, R¹, and X⁽⁻⁾ have the same meanings as before; and

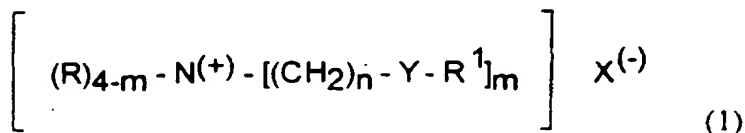
3. mixtures thereof;

- B. from about 10% to about 30% of said high boiling, water soluble organic solvent;
- C. from 0% to about 10%, of low molecular weight alcohol selected from the group consisting of: ethanol, isopropanol, n-propanol, n-butanol, t-butanol alcohol, and mixtures thereof;
- D. from 0% to about 15% perfume; and
- E. optionally, from about 2% to about 35% of dispersing aid.

3. The fabric softener premix composition of Claim 2 comprising:

- A. from about 65% to about 75% of said fabric softener active said fabric softener active being:

1. fabric softener compound having the formula:



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- 10 wherein each R substituent is a short chain C₁-C₃ alkyl or hydroxyalkyl group, benzyl or mixtures thereof; each m is 2; each n is from 2 to about 3; for each R¹ the Iodine Value of the corresponding fatty acid is from about 70 to about 105;
- 15 B. from about 12% to about 25% of said high boiling water soluble organic solvent;
- C. optionally, from 0% to 6%, and sufficient to improve viscosity, of said low molecular weight alcohol;
- D. optionally, from about 1% to about 10% perfume; and
- E. optionally, from about 3% to about 25% of said dispersing aid.
4. The fabric softener premix composition of Claim 3 containing:
- 5 A. from about 65% to about 75% by weight of the premix composition, of said fabric softener wherein each n is 2; each R¹ is a long chain C₁₃-C₁₇ straight chain alkyl or alkylene, the total level of polyunsaturated acyl groups, comprising R¹ is from about 5% to about 18%, and the cis/trans ratio is from about 1:1 to about 50:1;
- 10 B. from about 13% to about 20% of said high boiling, water soluble organic solvent;
- C. optionally, from 0% to about 6%, and sufficient to improve viscosity, of said low molecular weight alcohol; and
- D. optionally, from about 1% to about 5% by weight of the premix composition of said perfume;
- E. optionally, from about 4% to about 15% of said dispersing aid.
5. The fabric softener premix composition of Claim 1 wherein the softener active contains up to about 20% of monoester compound wherein m is 2, and YR¹ is either H or -C(O)OH.
6. Finished fabric softener composition comprising the premix composition of Claim 1 wherein the level of high boiling water soluble solvent B. is sufficient to provide the required viscosity without rendering the composition flammable.
7. The finished fabric softener composition of Claim 6 wherein the desired viscosity is less than about 1000 cps at room temperature.

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8. The finished fabric softener composition of Claim 6 wherein the desired viscosity is less than about 500 cps at room temperature.

9. The process of making a finished fabric softening composition by adding the premix composition of Claim 1 to a water seat comprising water; acid to create a pH of from about 1.5 to about 5; and, optionally, an effective amount of water soluble electrolyte, and mixing.

10. The process of making a finished fabric softening composition by adding the premix composition of Claim 2 to a water seat comprising water; acid to create a pH of from about 1.5 to about 5; and, optionally, an effective amount of water soluble electrolyte, and mixing.

11. The process of making a finished fabric softening composition by adding the premix composition of Claim 4 to a water seat comprising water; acid to create a pH of from about 1.5 to about 5; and, optionally, an effective amount of water soluble electrolyte, and mixing.

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(21) International Application Number: PCT/US97/15122 (22) International Filing Date: 28 August 1997 (28.08.97) (30) Priority Data: 60/024,896 30 August 1996 (30.08.96) US (71) Applicant (for all designated States except US): THE PROCTER & GAMBLE COMPANY [US/US]; One Procter & Gamble Plaza, Cincinnati, OH 45202 (US). (72) Inventors; and (75) Inventors/Applicants (for US only): WAHL, Errol, Hoffman [US/US]; 8021 Deersadow Lane, Cincinnati, OH 45242 (US). TRINH, Toan [US/US]; 8671 Creekwood Lane, Maineville, OH 45039 (US). DEMEYERE, Hugo, Jean-Marie [BE/BE]; Linthout Straat 59, B-1785 Merchtem (BE). DECLERQ, Marc, Johan [BE/BE]; Ringlaan 77, B-1853 Strombeek-Bever (BE). (74) Agents: REED, T., David et al.; The Procter & Gamble Company, 5299 Spring Grove Avenue, Cincinnati, OH 45217 (US).		(81) Designated States: BR, CA, CN, JP, MX, US, European patent (AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE). Published <i>With international search report.</i> <i>Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.</i> (88) Date of publication of the international search report: 7 May 1998 (07.05.98)
(54) Title: CONCENTRATED PREMIX WITH REDUCED FLAMMABILITY FOR FORMING FABRIC SOFTENING COMPOSITION		
(57) Abstract Biodegradable fabric softener premix compositions are described containing (1) biodegradable softener actives; optional low molecular weight solvent; (2) high boiling, water soluble solvent to lower the viscosity, while avoiding flammability; and (3) optional perfume. The premixes can be added at ambient temperatures to water containing acid, to provide a pH of from about 1.5 to about 5, to form finished compositions. The water can also contain calcium and/or magnesium salt to modify the viscosity. Compositions containing highly unsaturated softener actives have improved freeze/thaw properties.		

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INTERNATIONAL SEARCH REPORT

International Application No
PCT/US 97/15122

A. CLASSIFICATION OF SUBJECT MATTER
IPC 6 C11D1/62 C11D3/43 //C11D3/50,C11D3/20

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 6 C11D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	DE 36 08 093 A (HENKEL KGAA) 17 September 1987 see example 2	1-4,9-11
A	see claims 1-6 ---	5-8
X	EP 0 644 179 A (STEPAN EUROP) 22 March 1995 see example 1	1,5
A	---	2-4,6-8
X	US 5 525 245 A (GRANDMAIRE JEAN-PAUL ET AL) 11 June 1996 see claims 1-3,15-20	1
A	---	2-11
X	US 5 474 690 A (WAHL ERROL H ET AL) 12 December 1995 see claims 25-33 ---	1
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12 March 1998

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INTERNATIONAL SEARCH REPORT

International Application No

PCT/US 97/15122

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT		
Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
P, X	WO 97 03172 A (PROCTER & GAMBLE) 30 January 1997 see claim 1	1
A	see page 76, line 5 - line 18 ---	2-11
A	DE 43 07 186 A (HENKEL KGAA) 15 September 1994 see page 3, line 4 - page 4, line 18 see claims 1-5 ---	1-11
A	EP 0 404 471 A (UNILEVER PLC ;UNILEVER NV (NL)) 27 December 1990 see page 3, line 37 - page 4, line 12 see page 7, line 47 - page 8, line 2 ---	1-11
E	WO 97 34976 A (PROCTER & GAMBLE) 25 September 1997 see claims 1-7 -----	1-11

INTERNATIONAL SEARCH REPORT

information on patent family members

International Application No

PCT/US 97/15122

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
DE 3608093 A	17-09-87	DK 121187 A EP 0240727 A JP 62223375 A	13-09-87 14-10-87 01-10-87
EP 0644179 A	22-03-95	AT 147719 T DE 69307507 D DE 69307507 T ES 2097476 T	15-02-97 27-02-97 15-05-97 01-04-97
US 5525245 A	11-06-96	AU 4525096 A CA 2208368 A CZ 9701925 A EP 0801672 A FI 972647 A NO 972874 A PL 321433 A WO 9619552 A US 5656585 A	10-07-96 27-06-96 12-11-97 22-10-97 18-08-97 13-08-97 08-12-97 27-06-96 12-08-97
US 5474690 A	12-12-95	CZ 9701417 A EP 0792335 A FI 972036 A NO 972192 A WO 9615212 A	17-09-97 03-09-97 13-05-97 28-05-97 23-05-96
WO 9703172 A	30-01-97	AU 6488996 A AU 6544396 A AU 6636596 A WO 9703169 A WO 9703170 A AU 2066597 A WO 9734972 A	10-02-97 10-02-97 10-02-97 30-01-97 30-01-97 10-10-97 25-09-97
DE 4307186 A	15-09-94	DE 59402536 D WO 9420596 A EP 0688356 A ES 2100703 T	28-05-97 15-09-94 27-12-95 16-06-97
EP 0404471 A	27-12-90	AU 623019 B AU 5711990 A	30-04-92 20-12-90

INTERNATIONAL SEARCH REPORT

information on patent family members

International Application No

PCT/US 97/15122

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
EP 0404471 A		CA 2018789 A	19-12-90
		DE 69019236 D	14-06-95
		DE 69019236 T	05-10-95
		ES 2072981 T	01-08-95
		JP 2562843 B	11-12-96
		JP 3027180 A	05-02-91
<hr/>			
WO 9734976 A	25-09-97	AU 2211597 A	10-10-97
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